

UNITED STATES PATENT OFFICE.

ADOLF ISRAEL AND RICHARD KOTHE, OF ELBERFELD, GERMANY, ASSIGN-
ORS TO FARBENFABRIKEN OF ELBERFELD CO., OF NEW YORK, N. Y.

AZO DYE.

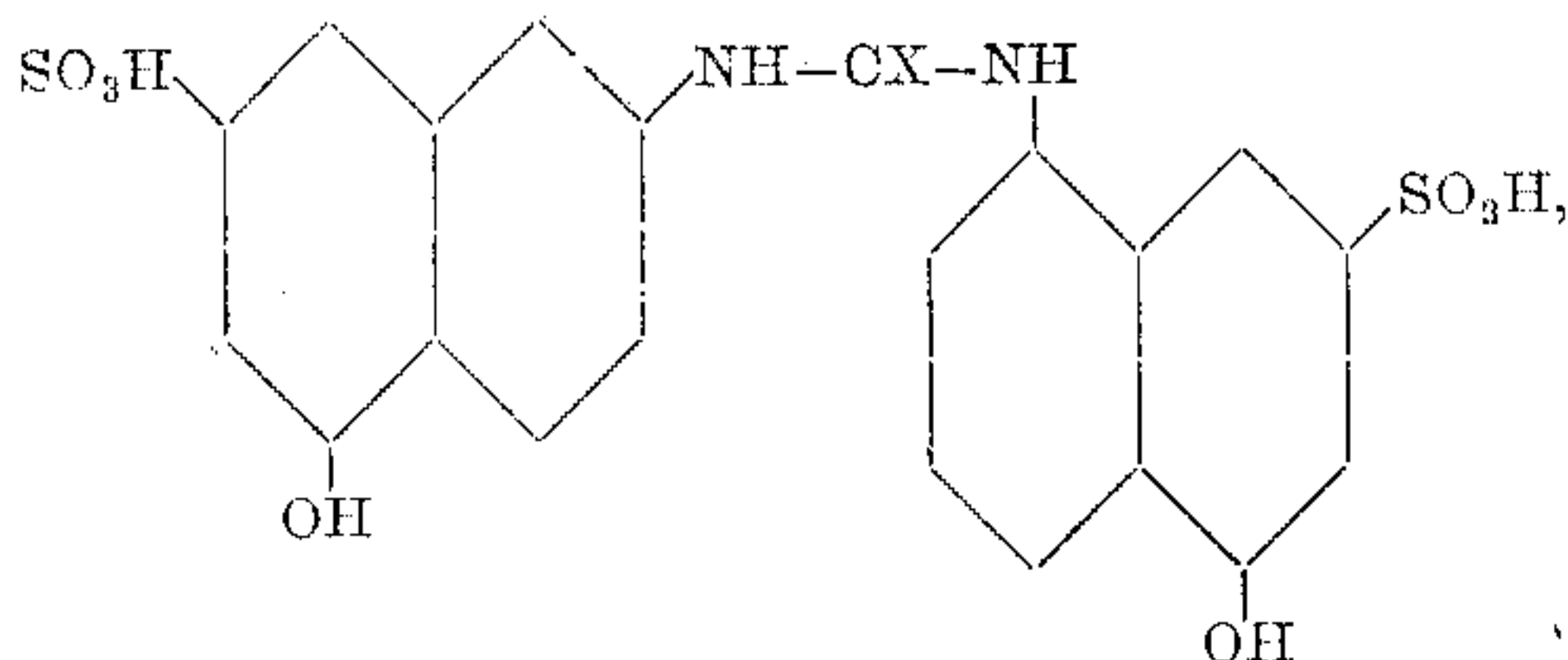
SPECIFICATION forming part of Letters Patent No. 675,632, dated June 4, 1901.

Application filed December 31, 1900. Serial No. 41,627. (Specimens.)

To all whom it may concern:

Be it known that we, ADOLF ISRAEL and RICHARD KOTHE, doctors of philosophy, chem-
ists, (assignors to the FARBENFABRIKEN OF ELBERFELD COMPANY, of New York,) residing
at Elberfeld, Germany, have invented a new
and useful Improvement in New Azo Dyes;
and we hereby declare the following to be a
clear and exact description of our invention.

In the two applications for Letters Patent
filed December 31, 1900, and bearing the Se-
rial Nos. 41,628 and 41,629, we have described
the production of new urea and thio-urea com-
pounds having the formula:

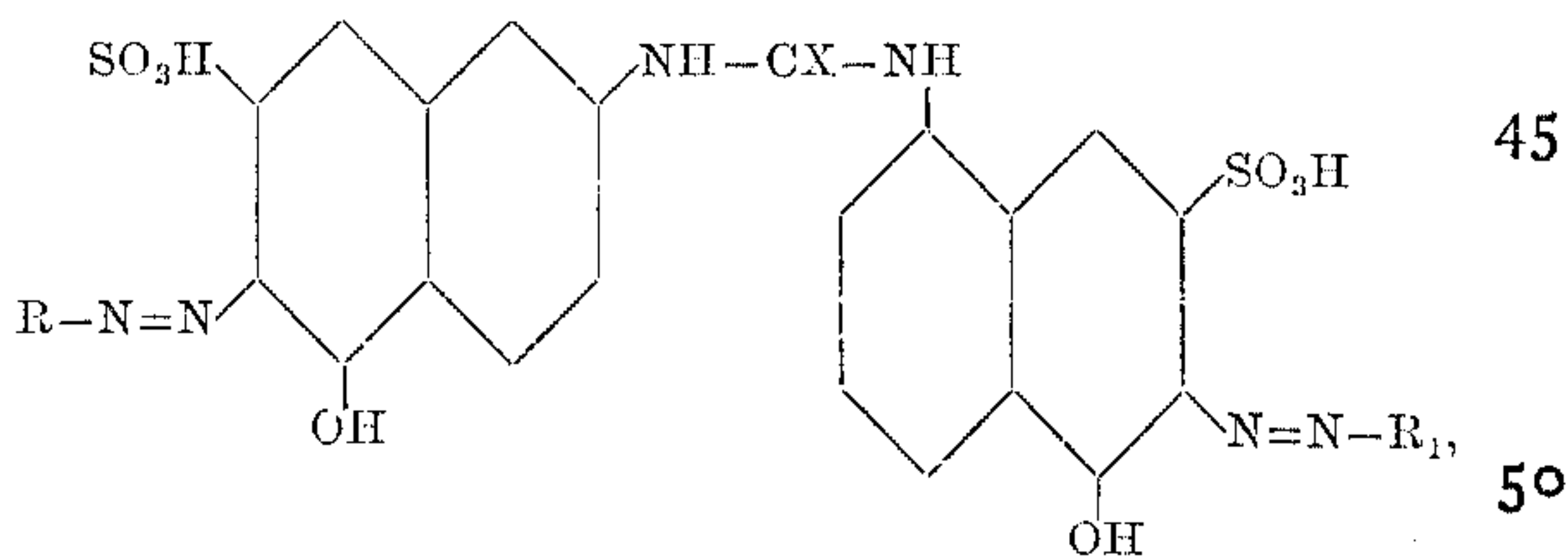


(in which formula X means an atom of oxy-
gen, which may be replaced by an atom of
sulfur.)

Our present invention relates to the pro-
duction of valuable new azo coloring-matters
by causing two molecules of the same or of
different diazo compounds to act on the said
urea or thio-urea compound.

We point out specifically that by the term
"diazo compound" in the following specifica-
tion we intend to denote a simple diazo com-
pound as well as a diazotized amidoazo com-
pound, such as diazoazobenzene, diazoazo-
toluene, or the like.

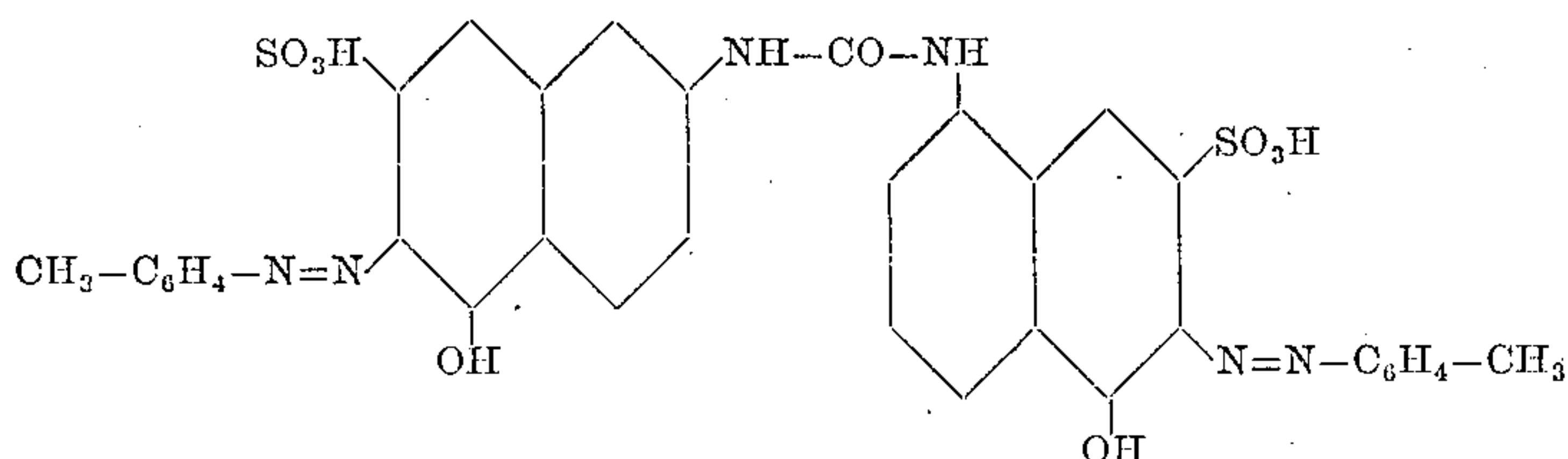
The new dyestuffs are alkaline salts of acids
having most probably the following general
formula:



(in which formula X means an atom of oxy-
gen, which may be replaced by an atom of sul-
fur, the groups $-N=N-R$ and $-N=N-R_1$
meaning either the same or two different radi-
cals of "diazo compounds"—such as diazo-
benzene, diazotoluene, diazoazobenzene, di-
azoazonaphthalene, or the like—and which
are from reddish-brown to dark-brown pow-
ders having a metallic luster, dissolving in wa-
ter with from orange to bluish-red color. They
dye unmordanted cotton from orange to blu-
ish-red shades, which are of a remarkable fast-
ness to light.)

In carrying out our new process practically
we can proceed as follows, the parts being by
weight: 15.3 parts of para-toluidin are diazo-
tized in the usual manner with the aid of
fifty-six parts of hydrochloric acid (of 15°
Baumé) and ten parts of sodium nitrite. The
resulting diazo solution is then slowly stirred
into a solution prepared from 39.2 parts of
the sodium salt of carbonyldioxydinaphthyl-
amindisulfonic acid, which is the scientific
name of one of the above-mentioned urea
compounds, (prepared from beta₁-amido-al-
pha₃-naphthol-beta₄-mono-sulfonic acid and
alpha₁-amido-alpha₃-naphthol-beta₄-sulfonic
acid,) and thirty parts of sodium carbonate,
(Na₂ CO₃.) After having stirred for several
hours the formation of the dyestuff has been
finished. The latter is separated from the
mixture by the addition of common salt and
isolated by filtration. The new coloring-mat-
ter thus obtained is the sodium salt of an acid
having most probably the formula:

Correction in Letters Patent No. 675,632.

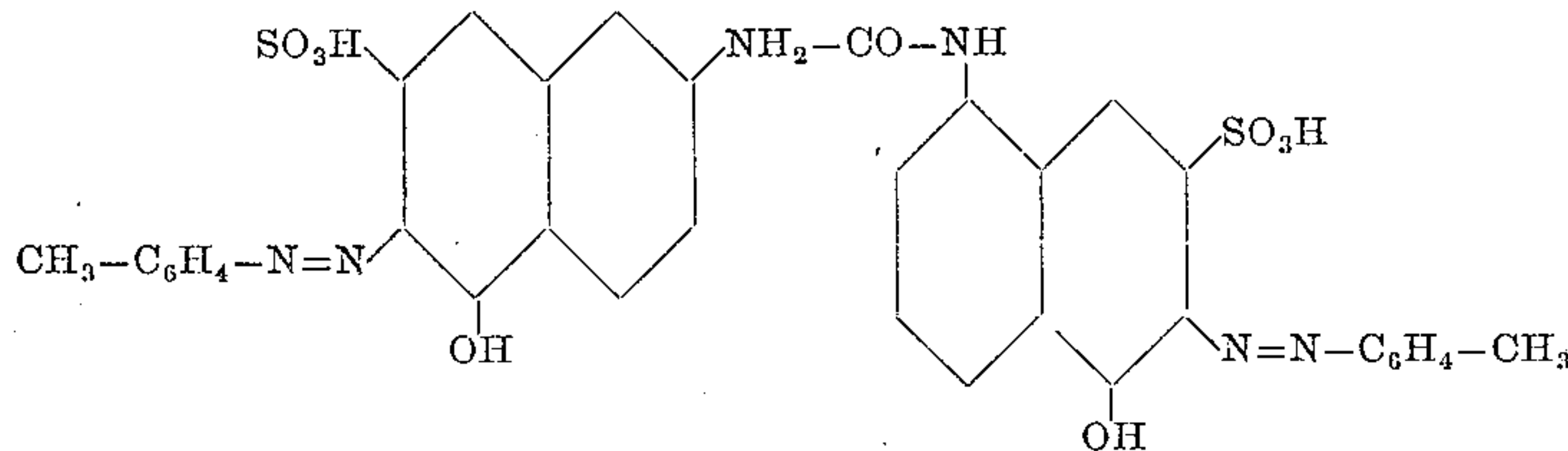


and is a reddish-brown powder having a metallic luster soluble in water with a red color. It is scarcely soluble in concentrated hydrochloric acid, while it is dissolved by concentrated sulfuric acid (of 66° Baumé) with a red color, which is changed into bluish red on the addition of a small quantity of ice, while on the addition of a larger quantity of ice a reddish-blue precipitate is obtained.

The new coloring-matter dyes unmordanted cotton red shades, which are fast to acids and to light.

The process proceeds in an analogous manner if instead of two molecules of diazotized paratoluidin other diazotized compounds are employed. If two different diazo compounds are employed, the first component is combined with the urea derivative in a slightly-acid and the second in an alkaline solution. On using, for instance, two molecules of diazotized beta-naphthylamin a bluish-red dyestuff is obtained. On using one molecule of diazotized anilin and one molecule of diazotized acetyl-para-phenylene-diamin also a bluish-red dyestuff is produced.

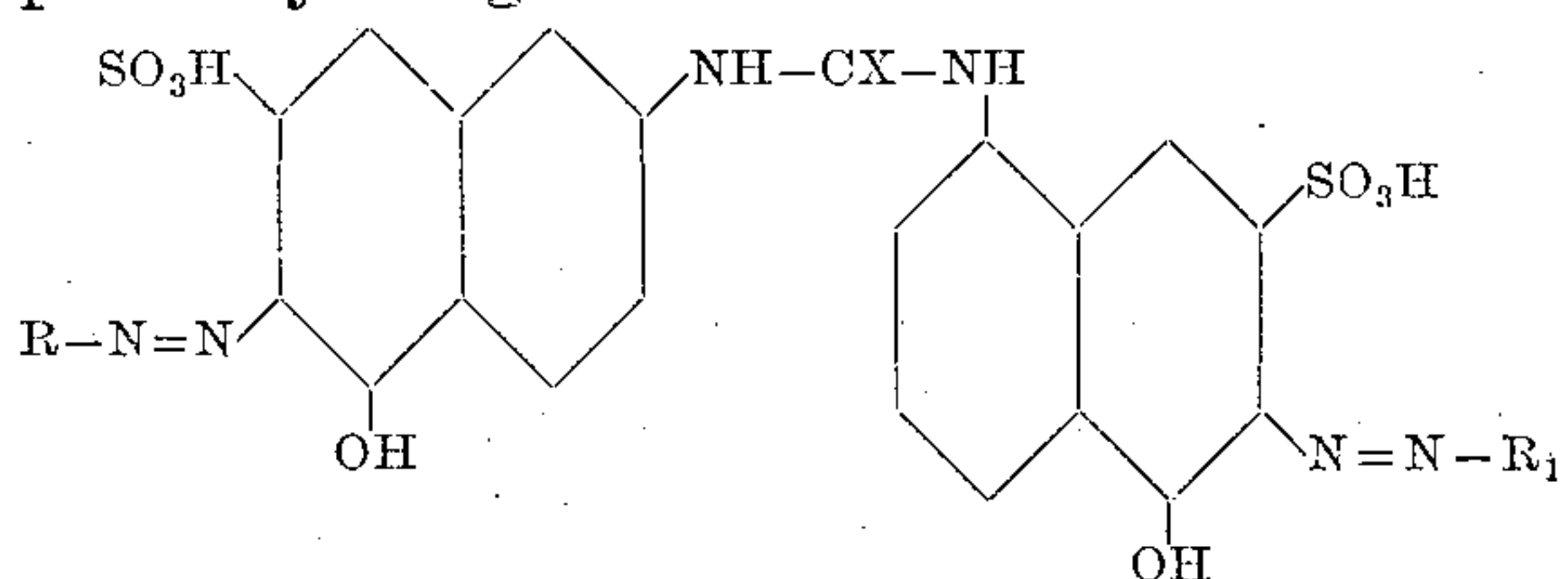
Having now described our invention and in what manner the same is to be performed,



which in the form of its sodium salt is a reddish-brown powder having a metallic luster, soluble in water with a red color; scarcely soluble in concentrated hydrochloric acid; being dissolved by concentrated sulfuric acid (of 66° Baumé) with a red color which is changed into bluish red on the addition of a small quantity of ice, while on the addition of a larger quantity of ice a reddish-blue precipitate is obtained, dyeing unmordanted cot-

ton red shades which are fast to acids and to light, substantially as hereinbefore described.

1. The herein-described new azo dyestuffs which are alkaline salts of acids having most probably the general formula:



X meaning in this formula an atom of oxygen which may be replaced by an atom of sulfur, the groups $-N=N-R$ and $-N=N-R_1$ meaning the radicals of "diazo compounds," and which are from reddish-brown to dark-brown powders having a metallic luster, soluble in water with from an orange to bluish-red color, dyeing unmordanted cotton from orange to bluish-red shades, which are fast to light, substantially as hereinbefore described.

2. The herein-described new disazo dyestuff being an alkaline salt of an acid, having most probably the formula:

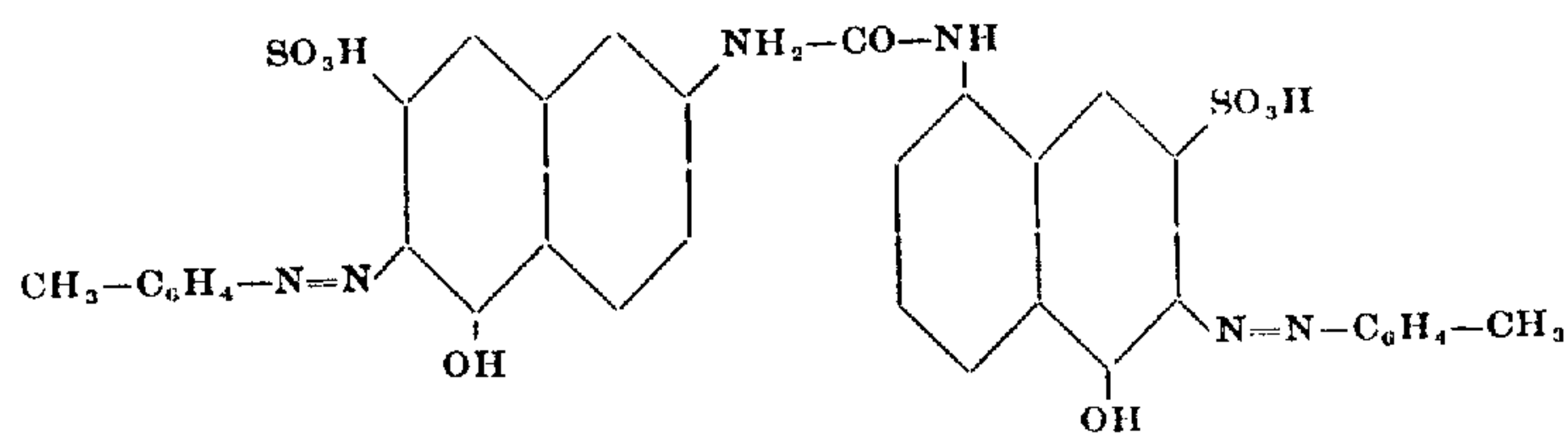
ton red shades which are fast to acids and to light, substantially as hereinbefore described.

In testimony whereof we have signed our names in the presence of two subscribing witnesses.

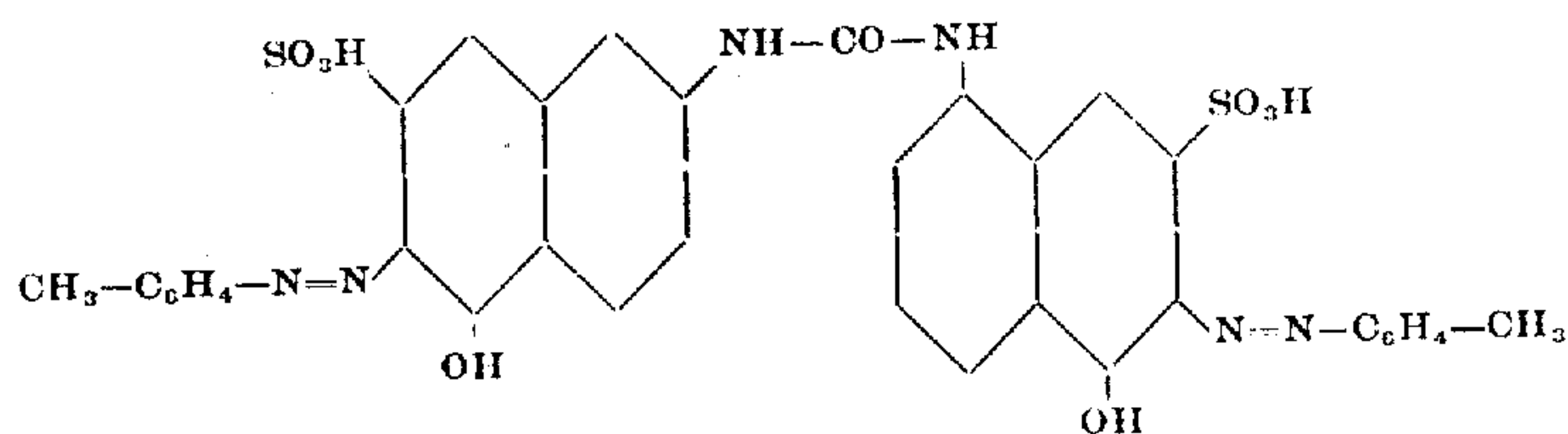
ADOLF ISRAEL.
RICHARD KOTHE.

Witnesses:
OTTO KÖNIG,
J. A. RITTERSHAUS.

It is hereby certified that in Letters Patent No. 675,632, granted June 4, 1901, upon the application of Adolf Israel and Richard Kothe, of Elberfeld, Germany, for an improvement in "Azo Dyes," an error appears in the printed specification requiring correction as follows: On page 2, the formula following line 54,



should read



and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed, countersigned, and sealed this 13th day of August, A. D., 1901.

[SEAL.]

F. L. CAMPBELL.
Assistant Secretary of the Interior.

Countersigned:

E. B. MOORE,
Acting Commissioner of Patents.